PATENT ABSTRACTS OF JAPAN

(11)Publication number : 06-025702

(43)Date of publication of application : 01.02.1994

(51)Int.CI.

B22F 1/00

B22F 9/22

G11B 5/62

H01F 1/047

(21)Application number : 04-183458

(22)Date of filing : 10.07.1992

(71)Applicant : KANTO DENKA KOGYO CO LTD

(72)Inventor : YOSHIDA ISAO
OBAYASHI NOBUO
IIZUKA SHINJI
MORIYA YOSHIMI
ENDO MITSURU

(54) MAGNETIC METAL POWDER AND ITS PRODUCTION

(57)Abstract:

PURPOSE: To produce a magnetic metal powder having high coercive force, saturation magnetization, etc., by coating α-FeOOH having a specified specific surface with a specified element, heat-treating the coated material in a nonreducing atmosphere and then reducing the material with a reducing gas.

CONSTITUTION: The α-FeOOH having 60-130m2/g specific surface is coated with specified amts. of Ni, Co, Al, Si and ≥1 kind of the compd. of rare-earth elements, heat-treated at about 300-800°C in a nonreducing atmosphere and then reduced at about 300-600°C in a gaseous hydrogen current. Consequently, a magnetic metal powder consisting essentially of iron and having 0.05-0.3μ major axis, 130-170Å crystal size, 45-70m2/g specific surface and ≥100emu/g saturation magnetization after standing for 7 days in the atmosphere kept at 60°C and having 90% relative humidity is obtained. The powder is excellent in dispersibility, packing property and oxidation stability and has a narrow coercive force distribution when formed into tape.

LEGAL STATUS

[Date of request for examination] 09.03.1999

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number] 3268830

[Date of registration] 18.01.2002

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

CLAIMS

[Claim(s)]

[Claim 1] nickel and Co And contain the compound of Si and aluminum in one or more sorts of rare-earth-element lists. the diameter of a major axis -- 0.05-0.3 the metal magnetism powder mum and whose microcrystal size are 130 - 170 **s, whose specific surface area is 45-70m2/g and which uses iron as a principal component -- it is -- coercive force (Hc) -- 1600 - 2000 Oe And metal magnetism powder characterized by the amount of saturation magnetization (sigmas) after putting on the ambient atmosphere of the temperature of 60 degrees C and 90% of relative humidity gently for seven days being 100 or more emu/g.

[Claim 2] specific surface area -- 60-130m2/g it is -- alpha-FeOOH -- nickel, Co, aluminum, and Si And the manufacture approach of the metal magnetism powder according to claim 1 characterized by making the compound of one or more sorts of rare earth elements put (however, the case where initial-complement content is carried out already being removed to alpha-FeOOH), heat-treating under a nonreducible ambient atmosphere, and subsequently returning by reducibility gas.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention is coercive force Hc and amount of saturation magnetization sigmas, though it is the particle optimal as raw material magnetism powder for magnetic-recording media which needs high power in the object for high density magnetic-recording media, especially a short wavelength field. Both, it is high, and excels also in dispersibility, packing nature, and oxidation stability, and is related with the metal magnetism powder with small coercive force distribution (S. F.D. value) and its manufacture approach when considering as a tape.

[0002]

[Description of the Prior Art] In recent years, metal magnetism powder is widely used as a magnetic material for high density magnetic-recording media as the metal tape for audios, the metal tape for 8 m/mVTR, DAT, and a business-use magnetic-recording tape. Recently, development of the equipment which aimed at the formation of small lightweight of a magnetic recording medium, long duration record, and high definition-ization is performed briskly, and high-performance-izing and high density record-ization are required more also for the magnetic tape which is the magnetic-recording medium to be used. That is, a high increase in power and low noise-ization are called for, and especially a high increase in power in a short wavelength field is desired. The

metal magnetism powder used for producing the magnetic-recording medium which satisfies these requirements must be detailed, and coercive force and its amount of saturation magnetization must be high, it must be excellent in dispersibility and packing nature, and must be excellent also in oxidation stability. On the other hand, an advance of the technique about the magnetic head for amelioration of the latest magnetic recording medium to perform magnetic recording, playback, and elimination also **** better ** and in it has a remarkable thing. The activity of the medium of the high coercive force which was not able to use development of the new construction material for the magnetic heads and an advance of a processing technique until now is also attained, and the magnetic powder used for the future medium for high density magnetic recording is asked more for the thing of high coercive force. However, the actual condition is that what has the sharp coercive force distribution (S. F.D) when having high coercive force which will be demanded increasingly from now on, the amount of high saturation magnetization, and dispersibility, and excelling in packing nature and oxidation stability moreover, and making it a magnetic-recording medium when the metal magnetism powder manufactured with the conventional technique was made into a detailed particle which is suitable for high density record is not yet obtained. In order to attain the high increase in power of a magnetic-recording medium, it is required for the coercive force and the amount of saturation magnetization of magnetic material to be used to be high, but when the coercive force of metal magnetism powder is generally closely related to the magnitude of a particle and a particle becomes fine, it is a difficult direction to make coercive force high. For example, since 1/2 or less [of record wavelength] is temporarily desirable [coercive force becomes high so that an axial ratio (the diameter of a major axis / diameter of a minor axis) is size, but] as for the die length of a particle in order to obtain high power in a short wavelength field when the diameter of a minor axis of a needlelike particle is fixed, the die length of a particle will be mostly decided by record wavelength. On the contrary, when the die length of a particle is fixed, if the diameter of a minor axis is made small, an axial ratio becomes large and can make coercive force high, but when the diameter of a minor axis becomes not much small, it is known that superparamagnetism is discovered and coercive force ceases to be shown, and there is a limitation also in the diameter of a minor axis. Moreover, the amount of saturation magnetization is also closely related to the magnitude of a metal magnetism powder particle. Since it cannot be dealt with to stability in atmospheric air, the oxide film of 20-30A of thickness is made to form on the surface of a particle, and, moreover, it is known for the metal [having returned the metal magnetism powder used now] that the contribution to the amount of saturation magnetization of this oxide film is small. Moreover, even if a particle becomes fine, the same thickness as a large particle is required for the thickness of this oxide film. Therefore, if the particle is made fine, the rate of the oxide film occupied to magnetic material will become large, and with the metal magnetism powder of the same presentation, when a particle becomes fine, the amount of saturation magnetization will become small.

[0003] On the other hand, it is known that it is effective in amelioration of the noise of a magnetic-recording medium to make a magnetic material particle detailed. That is, generally it is known by making [many] the particle number included in per unit area of a magnetic-recording medium that reduction of a noise can be attained. There is specific surface area as one of the approaches showing the magnitude of a particle, and the specific surface area of a magnetic material particle and the relation of a noise are reported (for example, drawing 44 in corporation Japan magnetic media Semiconductor Equipment & Materials International issuance and magnetic media engineering manual No.3 "the principle of magnetic recording", 5). If the specific surface area of magnetic material becomes large, the noise is decreasing linearly, but since a problem is produced to distribution and packing nature if specific surface area becomes not much large, it may not be desirable to enlarge specific surface area. As another view, the microcrystal size of metal magnetism powder and the relation of a noise are also known, and a noise becomes small, so that microcrystal size is small. Therefore, they are detailed particles, and the requirements which make a noise small have a large specific surface area to extent which does not have an adverse effect on distribution and packing nature, and are small.

[of microcrystal size] Since the oxidation stability of metal magnetism powder is related to the preservation stability of a magnetic-recording medium, amelioration more extensive than the metal magnetism powder by which the current activity is carried out is desired. Assessment of oxidation stability carries out fixed period gentle placement of the metal magnetism powder into the elevated temperature and the highly humid ambient atmosphere, measures the amount of saturation magnetization after taking out, and is performed as compared with the amount of saturation magnetization before a test. If a metal magnetism powder particle becomes fine, it will become that surface activity generally increases and it is easy to oxidize, and stability will tend to worsen. Moreover, it is thought that the dispersibility of metal magnetism powder and packing nature change with that the configuration of a powder particle and a particle front face are chemical and a physical property. About the configuration, it is supposed that a monodisperse particle without branch separation, a hole, etc. is desirable. However, dispersibility and packing nature become difficult, so that it becomes a particle.

[0004]

[Problem(s) to be Solved by the Invention] With high-performance-izing of a magnetic recorder and reproducing device in recent years, the high-increase-in-power [of the magnetic-recording medium to be used], reduction in noise, and metal magnetism powder which want of a high increase in power in a short wavelength field is stronger than especially that of record wavelength, and is used is also detailed, coercive force is high, and the amount of saturation magnetization is also large, and what is excellent in dispersibility, packing nature, and oxidation stability is desired. However, in a Prior art, metal magnetism powder with which are satisfied of these items is not obtained. This invention aims at solving this problem.

[0005]

[Means for Solving the Problem] The following this inventions can attain the problem of said conventional technique.

[0006] This invention is nickel and Co in the first place. And contain the compound of Si and aluminum in one or more sorts of rare-earth-element lists. the diameter of a major axis -- 0.05-0.3 the metal magnetism powder mum and whose microcrystal size are 130 - 170 **s, whose specific surface area is 45-70m2/g and which uses iron as a principal component -- it is -- coercive force (Hc) -- 1600-2000Oe And it is the metal magnetism powder characterized by the amount of saturation magnetization (sigmas) after putting on the ambient atmosphere of the temperature of 60 degrees C and 90% of relative humidity gently for seven days being 100 or more emu/g. the second specific surface area -- 60-130m2/g it is -- alpha-FeOOH -- nickel, Co, aluminum, and Si And it is the manufacture approach of the above-mentioned metal magnetism powder characterized by making the compound of one or more sorts of rare earth elements put (however, the case where initial-complement content is carried out already being removed to alpha-FeOOH), heat-treating under a nonreducible ambient atmosphere, and subsequently returning by reducibility gas.

[0007] The metal magnetism powder of this invention uses as a start raw material the oxy-iron hydroxide obtained by blowing a oxidizing gas into the water suspension which mixed alkali with ferrous salt. As a class of this oxy-iron hydroxide, alpha-FeOOH is desirable, hydroxylation alkali neutralizes ferrous salt as that process, and it is Fe (OH)2. There is the first process which uses as water suspension, blows a oxidizing gas into this suspension and is made into needlelike alpha-FeOOH. On the other hand, carbonic acid alkali neutralizes ferrous salt and it is FeCO3. There is the second process which uses as water suspension, blows a oxidizing gas into this suspension and is made into fusiform alpha-FeOOH. As ferrous salt used by these approaches, any of ferrous chloride, the first iron of a nitric acid, and a ferrous sulfate may be used. Moreover, as hydroxylation alkali used by the first process, a potassium hydroxide, a sodium hydroxide, and aqueous ammonia are usable. Moreover, as carbonic acid alkali, a sodium carbonate, sodium bicarbonate, an ammonium carbonate, etc. are usable. The conditions which manufacture alpha-FeOOH suitable for obtaining the metal magnetism powder which is detailed, does not have branching and is excellent in dispersibility and packing nature using the first process of the above are using the amount of two to 10 times of the amount of alkali required for neutralization

of ferrous salt, and advancing oxidation reaction of Fe (OH)2 on the conditions alkali concentration's being high. The reactions in the field where alkali concentration is high are conditions required to obtain a particle without branching. moreover -- although control of the magnitude of a particle is possible at control of reaction temperature and the amount of entrainments of a oxidizing gas as generally known -- in addition, the inside of ferrous salt -- nickel, Co, aluminum, and Si etc. -- after it makes the metal salt exist and alkali neutralizes this, advancing oxidation reaction can also control the dimension of a particle In the second process, fusiform alpha-FeOOH to generate does not have branching and the detailed particle to which grain size was equal tends to obtain it. Moreover, control of the magnitude of the particle in the second process is possible by changing the concentration of the iron in water suspension, reaction temperature, and the amount of entrainments of a oxidizing gas. moreover, the first process -- the same -- nickel and Co etc. -- the configuration of a particle is controllable with addition.

[0008] The manufacture approach of the metal magnetism powder of this invention which uses as a raw material hereafter needlelike alpha-FeOOH obtained by the first process is explained as an example. First, the hydroxylation alkali more than the amount of 2 double of a complement neutralizes ferrous salt to neutralization, and it is Fe (OH)2. It considers as alkali suspension, a oxidizing gas is blown into this, and needlelike alpha-FeOOH is obtained. At this time, in order to control the needlelike ratio and configuration of alpha-FeOOH, metals, such as nickel, Co, Zn, Cr, Mn, Zr, aluminum, Si, P, Ba, calcium, Mg, Cu, Sr, Ti, Mo, Ag, and a rare earth element, can be doped. You may mix to homogeneity in ferrous salt, and the addition approach of these dissimilar metals may be added in the middle of a reaction. About an addition, it is experientially decided with the configuration and magnitude for which it asks. In addition, by this approach, alkali neutralizes ferrous salt, and it is Fe (OH)2. Although suspension is made to generate, this is oxidized and alpha-FeOOH is manufactured, when it considers as metal magnetism powder by using the amount of alkali used at this time more than twice [at least] rather than a neutralization equivalent, high raw material alpha-FeOOH of coercive force is obtained. The more there are many excessive amounts of such alkali, the more branching of alpha-FeOOH decreases, but since the effectiveness beyond it does not discover even if it adds 10 or more times, the reaction which applies the excessive amount of 10 times or more is not efficient. Moreover, for the magnitude of an alpha-FeOOH particle required to obtain the metal magnetism powder of this invention, the specific-surface-area value is 60-130m2/g. To consider as the magnitude used as the range is required. A particle is too large, this specific surface area is not obtained and high coercive force does not have it as a magnetic material for single wavelength fields at below 60m2/g. [desirable] Moreover, specific surface area is 130m2/g. Above, it will become what high coercive force is not acquired [whether does a particle become fine too much and superparamagnetism is discovered and], and is depended irregularly [a particle], or the large thing of coercive force distribution. Whether nickel, Co, Zn, Cr, Mn, Zr, aluminum, Si, P, Ba, calcium, Mg, Cu, Sr, Ti, Mo, Ag, a rare earth element, etc. are doped next, to or alpha-FeOOH which is not doped nickel, Co, aluminum, and Si And although one or more sorts of a rare earth element are made to contain, the approach of neutralizing various metal salts with an acid or alkali, and putting as film of the minute crystal of a hydroxide on a particle front face as an approach of making it contain, is common. Since nickel, Co, and a rare earth element have a limitation in the amount doped when it is necessary to make these elements contain so much, they make these elements put on a front face further, although it is not necessary to make the front face of an alpha-FeOOH particle newly cover when the initial-complement dope is carried out at the reaction which makes alpha-FeOOH generate. In addition, the content of each metallic element in metal magnetism powder has the following desirable range. The following numeric values are iron 100 It is the weight ratio of each metal when carrying out.

0.5 to nickel=0.3-8.0Co=3.0-45.0aluminum=0.5 - 8.0Si=8.0 rare earth element = 0.2-10.0, however 2.0 to aluminum+Si=15.0 rare earth element are at least one kind in La, Ce, Pr, Nd, Sm, Gd, Dy, and Y, and are effective also in these combination. The metal to add has the convenient activity of water-soluble salts, such as

a chloride, a sulfate, and a nitrate. In the case of Si, meta-silicate of soda, orthosilicic acid soda, and water glass can be used. As for the sequence of covering, it is desirable to put previously nickel and Co which alloy first and control the magnetic properties of metal magnetism powder, and to put aluminum and Si which prevent sintering of the particle by heat next in the second half. In the case of a rare earth element, when putting nickel and/or Co, whether it dopes when generating an alpha-FeOOH particle, or it covers simultaneously, there is effectiveness which heightens coercive force. Although it is effective even if it covers, when putting aluminum and/or Si, made it larger [effectiveness] to exist in the interior.

[0009] Next, after carrying out specified quantity covering of each above-mentioned metal, this is fully rinsed, and it dries and heat-treats at the temperature of 300 - 800 ** in a nonreducible ambient atmosphere. The hole in the alpha-Fe 2O3 particle which alpha-FeOOH dehydrated and heat treatment temperature produced below in 300 ** will increase, consequently the property of the metal magnetism powder after reduction will be inferior. Moreover, at the temperature more than 800 **, fusion of 2Oalpha-Fe 3 particle starts [heat treatment temperature], the configuration of a particle changes, or sintering advances, and the property of the metal magnetism powder obtained as a result deteriorates. Next, return the metal magnetism powder after heat treatment at the temperature of 300 - 600 ** under a hydrogen gas air current, an oxide film is made to form on the surface of a particle by the well-known approach, and metal magnetism powder is obtained.

[0010]

[Function] The metal magnetism powder of this invention is the diameters 0.05-0.3 of a major axis. Though it is detailed, coercive force is [mum, microcrystal size 130 -170 ** and specific-surface-area / of 45-70m / 2/g, and] 1600 - 2000 Oe. It is high and the amount of saturation magnetization is also high, and the amount of saturation magnetization after putting on the ambient atmosphere of the temperature of 60 degrees C and 90% of relative humidity gently for seven days is excellent in 100 or more emu/g and oxidation stability, and excellent also in dispersibility and packing nature. although the metal magnetism powder which contains Co and a rare earth element, and aluminum or Si also in a Prior art (for example, JP,62-44842,B) is proposed -- the metal magnetism powder of this invention -- the metal magnetism powder of the conventional proposal -- comparing -- large -- a property -- improving . The technical difference with the proposal concerned is in this invention to have made addition of nickel, and coexistence of aluminum and Si into the indispensable condition. The effectiveness of addition of nickel is work which promotes the reduction for Fe in the multiple oxide of the difficulty reducibility generated when heat-treating alpha-FeOOH containing aluminum, Si, a rare earth element, etc. By this, aluminum and Si which are effective in preventing sintering at the time of reduction, and a rare earth element can be used so much. If there are not much few additions of nickel, since a reduction facilitatory effect reduces coercive force and the amount of saturation magnetization if it is not acquired and is not much abundant, it is not desirable. Coexistence of aluminum and Si is high coercive force, and although the metal magnetism powder which is excellent in dispersibility and packing nature and is excellent also in oxidation stability is obtained, it is indispensable. That is, although the metal magnetism powder of high coercive force is not obtained probably because an aluminum independent is inadequate as for the sintering prevention effectiveness, what is excellent in opposite side dispersibility and packing nature, and is excellent also in oxidation stability is obtained. On the other hand, although it is contrary to an aluminum independent case and easy to acquire high coercive force in an Si independent, that in which dispersibility, packing nature, and oxidation stability are a little inferior as compared with the case of aluminum is obtained. Therefore, by making aluminum and Si live together, both good points appear and what is excellent in dispersibility, packing nature, and oxidation stability with high coercive force is obtained. Furthermore, since a rare earth element has the sintering prevention effectiveness, when it contains this, its effectiveness improves further rather than the case of only aluminum and Si. The configuration of a particle holds the configuration of raw material alpha-FeOOH well, and what has small microcrystal size is obtained by high coercive force and the angle-of-elevation mold ratio by this. Although the reason which discovers such effectiveness is not clear, the

front face of the obtained metal magnetism powder particle is smooth as compared with what did not add a rare earth element. Since there are few holes and particles which broke few again, when an alpha-FeOOH front face is covered, the film more precise than the case of aluminum or Si generates, and it is thought that it can follow in footsteps of the volume cutback of a skeleton particle also in the reaction accompanied by volume changes, such as heat treatment and reduction. As stated above, the metal magnetism powder of this invention is high coercive force suitable as magnetic material for high density magnetic-recording media, the amount of high saturation magnetization, and small microcrystal size, and is excellent in dispersibility, packing nature, and oxidation stability.

[0011]

[Example] Although an example is given to below and this invention is explained to it in more detail, this invention is not limited to these.

To the container which passed example-1 nitrogen gas and drove out the oxidizing gas, they are 4l. of ferrous chloride water solutions of 2.0 mol/l, and 0.23 mols. The nickel chloride was mixed well, and it mixed, stirring 20l. of sodium-hydroxide water solutions of 2.5 mol/l to this, and suspension was obtained. Next, 10l. the air for /was blown for 2 hours, holding this suspension at 40 degrees C, and needlelike alpha-FeOOH by which nickel was doped was obtained. When the specific surface area of this thing was measured, it was 75m2/g (table 1 reference). Next, having made it distribute in 40l. distilled water, having considered as suspension, and stirring this suspension, after filtering the suspension containing alpha-FeOOH and rinsing the obtained residue (alpha-FeOOH), 0.76 mols of cobalt chlorides and the liquid which dissolved 0.065 mols of lanthanum chlorides in 2l. distilled water were added gradually, and was stirred with for [sufficient] 30 minutes. After churning and into this suspension, the sodium-hydroxide water solution was added gradually and pH of suspension was adjusted 8. Subsequently, in this suspension, the water solution which dissolved the 0.5-mol aluminum chloride in 2l. of distilled water was added, and it stirred with for [sufficient] 30 minutes. Furthermore to this suspension, it is 13.4g as a part for Si. After adding the included water glass water solution gradually, although pH of suspension is set to 8, the sodium-hydroxide water solution of a complement was added, and it fully stirred to it. Next, this suspension was filtered, the obtained residue was rinsed, and it dried in the oven of 120 **, and considered as the desiccation cake. Furthermore, this desiccation cake was heat-treated by 600 ** in nitrogen-gas-atmosphere mind for 2 hours, and, subsequently it returned by bottom of hydrogen gas stream 480 ** for 4 hours. After changing gas into nitrogen gas after reduction and lowering temperature to a room temperature, into the nitrogen gas stream, air was passed gradually and stabilizing treatment was performed with the conventional method. As a result of measuring the diameter of a major axis and microcrystal size, specific surface area, and magnetic properties about the metal magnetism powder obtained by the above actuation, it is 0.24 micrometers of diameters of a major axis. They were microcrystal size 158A and specific-surface-area 55.6m2/g, coercive force Hc 1783 Oe, and amount of saturation magnetization sigmas 128.4 emu/g. Moreover, when the particle of this metal magnetism powder was observed with the transmission electron microscope, there was no sintering and there were few holes also in a particle. In addition, the measuring method of each above-mentioned measured value is as follows. It measures using the so-called nitrogen measuring method, and specific surface area is coercive force Hc and amount of saturation magnetization sigmas at the conditions of 0.85g/cc of **** about magnetic properties. It measured. Moreover, it is 200 at random out of the photograph of the metal magnetism powder photoed with the transmission electron microscope about the diameter of a major axis of a powder particle. The particle of an individual is extracted and they are these [200]. The diameter of an average major axis of the particle of an individual showed. It is Fe (110) obtained with X-ray diffractometer about microcrystal size. It asked by the degree type from the half peak width of a diffraction peak, and 2theta value.

D (110) = Wavelength beta of a K lambda/beta cos thetaK; SHIERA constant (0.9) lambda; exposure X-ray; half peak width of a diffraction peak (it amends and uses for a true value)

theta; in order to evaluate the oxidation stability of an angle of diffraction and the metal magnetism powder obtained by this example, after putting this metal magnetism powder for seven days into the ambient atmosphere of the temperature of 60 degrees C, and 90% of relative humidity, that amount of saturation magnetization was measured. A result is shown in a table 2. Moreover, in order to evaluate the magnetic properties at the time of using the metal magnetism powder obtained by this example as magnetic material of the magnetic tape for high density magnetic-recording media and dispersibility, and packing nature, while tape-izing according to the actuation shown below and measuring those magnetic properties, dispersibility and packing nature were evaluated from the numeric value and square shape ratio of Br of this tape-izing. Moreover, coercive force distribution of metal magnetism powder was evaluated from the S.F.D. value of a tape. It mixed with 100g of approach metal magnetism powder and 11g of polyurethane resin of tape-izing, 7.75g of vinyl chloride vinyl acetate copolymers, and 345g of mixed solutions of toluene / methyl-ethyl-ketone / cyclohexanone =1/1/1, the Sand grinder performed distribution for 5 hours, and the magnetic coating was produced. After adding 2.5g coronate-L (product made from Japanese Polyurethane) as a cross linking agent in these coatings, it applied to polyester film, the 3000 gauss field was impressed, and it dried at the temperature of 50 degrees C. Subsequently, after performing calender processing by 80 degrees C and linear pressure 200 kg/cm and performing aging at 60 degrees C to the film after desiccation for 24 hours, it cut in the shape of a tape. Next, magnetic properties were measured by maximum impression magnetic field 5kOe about the obtained tape using the VSM magnetometer. A result is shown in a table 3.

[0012] To the container which passed example-2 nitrogen gas and drove out the oxidizing gas, they are 4l. of 2.0 mols [/l.] ferrous chloride water solutions, and 0.12 mols. The nickel chloride was mixed well, and it mixed, stirring 20l. of 2.5 mols [/l.] sodium-hydroxide water solutions to this. After adding the amount which considers water glass as a part for Si at this suspension, and is set to 0.9g and stirring, 10l. the air for /was blown for 2 hours, holding this suspension at 40 degrees C, and needlelike alpha-FeOOH of the particle by which nickel and Si were doped was obtained. When the specific surface area of this thing was measured, it was 92m2/g (table 1 reference). Next, distributing 40l. distilled water, considering as suspension, and stirring this suspension, after filtering the suspension containing above-mentioned alpha-FeOOH and rinsing the obtained residue A 1.5-mol cobalt chloride and 0.12 mols A nickel chloride and 0.09 mols The water solution which dissolved chlorination neodium in 2l. of distilled water was added, it stirred with for [sufficient] 30 minutes, the sodium-hydroxide water solution after churning was added gradually, and pH of suspension was adjusted to 8. Subsequently, the water solution which dissolved the 0.5-mol aluminum chloride in 2l. of distilled water was added to the above-mentioned suspension, it stirred with for [sufficient] 30 minutes, and the water glass water solution which contains 5.8g in this suspension as a part for Si was added further gradually. Next, although pH of suspension is set to 8, the sodium-hydroxide water solution of a complement was added, and it fully stirred to it. It processed like the example -1 hereafter, and metal magnetism powder was obtained. Moreover, oxidation stability was evaluated like the example -1 about the obtained metal magnetism powder in measurement of the diameter of a major axis and microcrystal size, specific surface area, and magnetic properties, and a list. A result is shown in a table 2. Moreover, the magnetic properties at the time of using the metal magnetism powder obtained by this example as magnetic material of the magnetic tape for high density magnetic-recording media etc. were evaluated like the example -1. A result is shown in a table 3.

[0013] An example -3 - 6 alpha-FeOOH are manufactured 4 times on the almost same conditions as an example -2, and it is an example about each. - It was referred to as 3, 4, 5, and 6, and specific surface area was measured about each. A result is shown in a table 1. Next, these examples - nickel, Co, aluminum, and Si which were used when performing covering processing to alpha-FeOOH of 3, 4, 5, and 6 Except having changed the amount, as shown in a table 1, and having changed the class of rare earth element to this table like the publication, it processes like an example -1 and is an example. - The metal magnetism powder of 3-6 was obtained. The acquired above-mentioned example - Oxidation stability was evaluated like the example -1 about the metal

magnetism powder of 3-6 in measurement of the diameter of a major axis and microcrystal size, specific surface area, and magnetic properties, and a list. A result is shown in a table 2. Moreover, example - The magnetic properties at the time of using as magnetic material of the magnetic tape for high density magnetic-recording media etc. were evaluated like the example -1 about the metal magnetism powder of an example 5 among the metal magnetism powder obtained by 3-6. A result is shown in a table 3. In the following examples and the example of a comparison, it was similarly estimated as the example -1.

[0014] Except having made into nickel and neodium the metal doped to example-7 alpha-FeOOH, it processed like the example -2 and metal magnetism powder was obtained.

Taught 4l. of ferrous chloride water solutions of 2.0 mol/l, and 20l. of 0.5 mols [/l.] sodium-carbonate water solutions to the container which passed example-8 nitrogen gas and drove out the oxidizing gas, often mixed, white iron carbonate was made to generate, and it considered as suspension. Stirring holding this suspension at 50 degrees C, 10l. the air for /was blown for 2 hours, and fusiform alpha-FeOOH was obtained. When the specific-surface-area value of this thing was measured, it was 85m2/g (table 1 reference). After filtering the suspension containing fusiform above-mentioned alpha-FeOOH and rinsing the obtained residue, 40l. distilled water was made to distribute this residue, and it considered as suspension, and in this suspension, stirring, it added gradually and the water solution which dissolved the 0.3 mols nickel chloride, the 0.76 mols cobalt chloride, and the 0.015-mol neodymium chloride in 3l. distilled water was stirred with for [sufficient] 30 more minutes. Subsequently, the sodium-hydroxide water solution was added and pH of suspension was adjusted to 8. Subsequently, the water solution which dissolved the 0.5-mol aluminum chloride in 2l. distilled water was added to this suspension, and it stirred with for [sufficient] 30 minutes to it. Furthermore to this suspension, it is 9g as a part for Si. After adding a water glass water solution gradually so that it may become an amount, the sodium-hydroxide water solution was added and pH of suspension was adjusted to 8. Next, this suspension was filtered, the obtained residue was rinsed, and it dried in the oven of 120 **, and considered as the desiccation cake. Next, this desiccation cake was heat-treated by 550 in nitrogen-gas-atmosphere mind ** for 2 hours, and, subsequently it returned by bottom of hydrogen gas stream 480 ** for 4 hours. After changing gas into nitrogen gas after reduction and lowering temperature to a room temperature, into the nitrogen gas stream, air was passed gradually and stabilizing treatment was performed with the conventional method. When the particle of this obtained metal magnetism powder was observed with the transmission electron microscope, it is 0.12 micrometers of diameters of a major axis. It was the particle which presented fusiform.

In the container with an agitator which passed example-9 nitrogen gas and drove out the oxidizing gas, the 1.5-mol cobalt chloride was often mixed with 4l. of ferrous-sulfate water solutions of 2.0 mol/l, and 20l. of sodium-carbonate water solutions of 0.7 mol/l and 1l. of sodium-hydroxide water solutions of 2 mol/l were added to this, and it stirred with for [sufficient] 30 minutes, and considered as suspension. Next, 10l. the air for /was blown for 2 hours, holding this suspension at 40 degrees C, and fusiform alpha-FeOOH by which cobalt was doped was obtained. The suspension containing fusiform above-mentioned alpha-FeOOH was filtered, the obtained residue was rinsed, after drying, the same processing as an example -8 was performed, and fusiform metal magnetism powder was obtained.

To the container with an agitator which passed example-10 nitrogen gas and drove out the oxidizing gas, they are 4l. of ferrous-sulfate water solutions of 2.0 mol/l, and 0.75 mols. The cobalt chloride was mixed well, and 20l. of ammonium-carbonate water solutions of 0.7 mol/l was added to this, and it stirred with for [sufficient] 30 minutes to it. Holding this suspension at 45 degrees C, 15l. the air for /was blown for 2 hours, and fusiform alpha-FeOOH by which cobalt was doped was obtained. The suspension containing fusiform above-mentioned alpha-FeOOH was filtered, the obtained residue was rinsed, after drying, it processed like the example -8 and fusiform metal magnetism powder was obtained.

Only by example -11 example -8 differing from the amount of addition metals, others were processed on the same conditions and obtained fusiform metal magnetism powder.

[0015] Except [all] having not added the lanthanum of example of comparison -1 rare earth element, it processed like the example -1 and needlelike metal magnetism powder was obtained. In addition, as compared with the metal magnetism powder obtained in the example -1, the metal magnetism powder of the example -1 of a comparison had low coercive force, and its microcrystal size was also large so that clearly also from a table 2 and a table 3.

Except [all] having not added example of comparison -2 cobalt, it processed like the example -2 and needlelike metal magnetism powder was obtained. In addition, as compared with the example -2, coercive force became low, the amount of saturation magnetization after putting for seven days into the temperature of 60 degrees C of metal magnetism powder and 90% of relative humidity became low, and the metal magnetism powder of the example -2 of a comparison had inadequate oxidation stability so that clearly also from a table 2 and a table 3.

Except having not added example of comparison -3 nickel, it processed like the example -8 and fusiform metal magnetism powder was obtained. In addition, the metal magnetism powder of the example -3 of a comparison became what has coercive force, the low amount of saturation magnetization, and a low square shape ratio as compared with the example -8 so that clearly also from a table 2.

Except [all] having not added example of comparison -4 rare earth element, it processed like the example -9 and fusiform metal magnetism powder was obtained. In addition, coercive force and a square shape ratio fell as compared with the example -9, microcrystal size was remarkable and the metal magnetism powder of the example -4 of a comparison had it so that clearly also from a table 2 and a table 3. [large]

Fusiform alpha-FeOOH of a raw material is manufactured by the same approach as example of comparison -5 example -8, and it processes, without adding Si, and considers as fusiform metal magnetism powder. In addition, as compared with the object which added Si, coercive force was remarkable and the metal magnetism powder of the example -5 of a comparison had it so that clearly also from a table 2. [low]

[0016] Fusiform alpha-FeOOH of a raw material is manufactured by the same approach as example of comparison -6 example -8, and it processes, without adding aluminum, and considers as fusiform metal magnetism powder. In addition, although coercive force became high as compared with what added aluminum so that clearly also from a table 2 and a table 3, it was that in which the dispersibility when making it powdered oxidation stability and a powdered tape is inferior.

[0017]
[A table 1]

添加金属の種類と添加量

No.	ドープ金属 種類と添加量	添加金属量（Feを100 とした時の重量比） Ni	Co	Al	Si	稀土類	α-FeOOHの 比表面積(m^2/g)
実施例－1	Ni=3.0	3.0	5.0	3.0	2.0	La=2.0	75.0
実施例－2	Ni=1.5 Si=0.2	3.0	20.0	3.0	1.5	Nd=3.0	92.0
実施例－3	Ni=1.5 Si=0.2	1.5	10.0	4.0	1.0	Dy=1.0	84.0
実施例－4	Ni=1.5 Si=0.2	5.0	10.0	2.5	2.5	Pr=1.0 Nd=1.0	90.0
実施例－5	Ni=1.5 Si=0.2	3.0	15.0	0.7	3.5	Nd=3.5	98.0
実施例－6	Ni=1.5 Si=0.2	3.0	15.0	4.5	0.7	Y =3.0	95.0
実施例－7	Ni=1.5 Nd=0.5	2.0	10.0	3.0	1.5	Nd=2.0	115.0
実施例－8		4.0	10.0	3.0	2.0	Nd=0.5	85.0
実施例－9	Co=20.0	5.0	40.0	2.0	1.5	Dy=3.5	90.0
実施例－10	Co=10.0	3.0	20.0	2.5	1.0	Nd=5.0	105.0
実施例－11		3.0	5.0	4.0	2.0	Nd=2.0	80.0
比較例－1	Ni=3.0	3.0	5.0	3.0	2.0	無添加	77.0
比較例－2	Ni=1.5 Si=0.2	3.0	無添加	3.0	1.5	Nd=3.0	92.0
比較例－3		無添加	10.0	3.0	2.0	Nd=0.5	86.0
比較例－4	Co=20.0	5.0	40.0	2.0	1.5	無添加	90.0
比較例－5		4.0	10.0	4.5	無添加	Nd=2.0	86.0
比較例－6		4.0	10.0	無添加	4.5	Nd=2.0	83.0

[0018]
[A table 2]

金属磁性粉末の磁気特性とその他特性

No.	磁気特性（測定磁場=10KOe）			平均長軸径（μm）	結晶子サイズ（Å）	比表面積（m^2/g）	高温高湿7日後のσs
	Hc(Oe)	σs(emu/g)	σr/σs				
実施例－1	1,783	128.4	0.520	0.24	158	55.6	102.8
実施例－2	1,926	145.7	0.535	0.17	163	57.4	115.0
実施例－3	1,628	136.5	0.515	0.16	157	58.5	110.5
実施例－4	1,820	135.0	0.523	0.14	161	60.4	107.1
実施例－5	1,964	139.2	0.540	0.18	167	54.5	106.4
実施例－6	1,651	137.4	0.516	0.15	155	62.3	111.5
実施例－7	1,718	132.8	0.518	0.11	158	58.0	111.4
実施例－8	1,720	134.0	0.498	0.12	147	59.5	108.6
実施例－9	1,825	149.3	0.502	0.08	165	48.7	121.8
実施例－10	1,684	142.3	0.500	0.10	156	58.5	111.0
実施例－11	1,625	126.8	0.496	0.14	143	60.3	104.3
比較例－1	1,520	133.5	0.492	0.21	172	52.5	98.5
比較例－2	1,708	122.5	0.502	0.18	158	58.8	84.7
比較例－3	1,587	124.7	0.477	0.13	155	62.4	92.4
比較例－4	1,494	158.7	0.469	0.07	196	43.5	120.7
比較例－5	1,460	135.1	0.472	0.11	140	52.0	108.8
比較例－6	1,890	131.4	0.505	0.14	145	54.3	88.5

[0019]
[A table 3]

テ ー プ 特 性

No.	テープの特性（測定磁場＝5kOe）			
	Hc(Oe)	Br(G)	角型比	S.F.D.
実施例－1	1,802	3,052	0.92	0.41
実施例－2	1,946	3,585	0.90	0.43
実施例－5	2,094	3,317	0.91	0.42
実施例－7	1,763	3,162	0.87	0.45
実施例－8	1,785	3,426	0.86	0.46
実施例－9	1,923	3,720	0.87	0.48
実施例－10	1,727	3,612	0.86	0.45
比較例－1	1,544	2,680	0.81	0.56
比較例－2	1,753	2,680	0.84	0.49
比較例－4	1,514	3,418	0.73	0.66
比較例－6	1,905	2,475	0.74	0.53

[0020]
[Effect of the Invention] It has the high coercive force required of the magnetic powder for high density magnetic-recording media though the metal magnetism powder of this invention is a particle, and the amount of high saturation magnetization, and excels in coercive force distribution (S. F.D.) and oxidation stability so that clearly in the comparison with the example and the example of a comparison which were described above, and it excels also in dispersibility further.

(19)日本国特許庁（ＪＰ）　(12)公開特許公報（Ａ）　(11)特許出願公開番号

特開平6－25702

(43)公開日　平成６年(1994)２月１日

(51)Int.Cl.[5]		識別記号	庁内整理番号	ＦＩ	技術表示箇所
Ｂ２２Ｆ	1/00	Ｗ			
	9/22	Ｃ			
Ｇ１１Ｂ	5/62		7303－5Ｄ		
Ｈ０１Ｆ	1/047				
				Ｈ０１Ｆ　1/06	Ｊ

審査請求　未請求　請求項の数２(全　９　頁)

(21)出願番号　特願平4－183458

(22)出願日　平成４年(1992)７月10日

(71)出願人　000157119
関東電化工業株式会社
東京都千代田区丸の内１丁目２番１号

(72)発明者　吉田　功
群馬県渋川市金井425番地　関東電化工業株式会社研究開発センター内

(72)発明者　大林　信夫
群馬県渋川市金井425番地　関東電化工業株式会社研究開発センター内

(72)発明者　飯塚　晋司
群馬県渋川市金井425番地　関東電化工業株式会社研究開発センター内

(74)代理人　弁理士　古谷　馨　(外３名)

最終頁に続く

(54)【発明の名称】　金属磁性粉末及びその製造方法

(57)【要約】

【目的】　微粒子でありながら、高密度磁気記録媒体用の磁性粉に要求される高保磁力、高飽和磁化量を有し、保磁力分布(S.F.D.)、酸化安定性に優れ、さらに分散性にも優れた金属磁性粉末を提供する。

【構成】　Ni,Co 及び１種以上の稀土類元素並びにSiとAlの化合物を含有する、長軸径が0.05～0.3 μｍ、結晶子サイズが130 ～170 Å、比表面積が45～70m²/gの、鉄を主成分とする金属磁性粉であって、保磁力(Hc)が1600～2000 Oe で、且つ温度60℃、相対湿度90％の雰囲気に７日間静置した後の飽和磁化量（σs)が100emu/g以上であることを特徴とする金属磁性粉末。

【特許請求の範囲】

【請求項１】　Ni,Co 及び１種以上の稀土類元素並びにSiとAlの化合物を含有する、長軸径が0.05～0.3 μm 、結晶子サイズが130 ～170 Å、比表面積が45～70m²/g の、鉄を主成分とする金属磁性粉であって、保磁力(Hc)が1600～2000 Oe で、且つ温度60℃、相対湿度90％の雰囲気に７日間静置した後の飽和磁化量（σs)が100emu/g以上であることを特徴とする金属磁性粉末。

【請求項２】　比表面積が60～130m²/g であるα-FeOOHにNi,Co,Al,Si 及び１種以上の稀土類元素の化合物を被着させ（但し、既にα-FeOOHに必要量含有されている場合を除く）、非還元性雰囲気下で熱処理し、次いで還元性ガスで還元することを特徴とする請求項１記載の金属磁性粉末の製造方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、高密度磁気記録媒体用、特に短波長領域で高出力を必要とする磁気記録媒体用の原料磁性粉として最適な、微粒子でありながら保磁力Hc、飽和磁化量σs ともに高く、且つ分散性、パッキング性、酸化安定性にも優れ、テープとしたときの保磁力分布（S.F.D.値）が小さい金属磁性粉末とその製造方法に関するものである。

【０００２】

【従来の技術】近年、金属磁性粉末は高密度磁気記録媒体用の磁性材料として、オーディオ用メタルテープ、８m/mVTR用メタルテープ、ＤＡＴ、及び業務用磁気記録テープとして広く使用されている。最近では磁気記録装置の小型軽量化、長時間記録、高画質化を目指した装置の開発が盛んに行われており、使用する磁気記録媒体である磁気テープも、より高性能化、高密度記録化が要求されている。即ち、高出力化と低ノイズ化が求められており、特に短波長領域での高出力化が望まれている。これらの要件を満たす磁気記録媒体を作製するのに用いられる金属磁性粉末は、微細であり、保磁力、飽和磁化量共に高く、分散性、パッキング性に優れ、酸化安定性にも優れるものでなければならない。一方、最近の磁気記録装置の改良は目ざましく、その中でも磁気記録、再生、消去を行うための磁気ヘッドに関する技術の進歩は著しいものがある。磁気ヘッド用の新材質の開発、加工技術の進歩は、今まで使用が不可能であった様な高保磁力の媒体の使用も可能になってきており、今後の高密度磁気記録用媒体に使用される磁性粉には、より高保磁力のものが求められている。しかるに、従来技術で製造される金属磁性粉末は、高密度記録に適するような微細な粒子にすると、今後益々要望されるような高保磁力、高飽和磁化量、および分散性を有し、しかもパッキング性、酸化安定性に優れ、且つ磁気記録媒体にしたときの保磁力分布（S.F.D)がシャープであるものは、未だ得られないのが現状である。磁気記録媒体の高出力化を達成するためには、使用する磁性材の保磁力及び飽和磁化量が高いことが必要であるが、金属磁性粉末の保磁力は一般には粒子の大きさに密接に関係しており、粒子が細かくなると保磁力を高くすることが難しい方向である。例えば、仮に針状粒子の短軸径が一定の場合、軸比（長軸径／短軸径）が大であるほど保磁力は高くなるが、短波長領域で高出力を得るためには粒子の長さは記録波長の１／２以下が好ましいので、粒子の長さは記録波長でほぼ決まってしまうこととなる。逆に、粒子の長さが一定の場合、短軸径を小さくしていけば軸比が大きくなり、保磁力を高く出来ることになるが、短軸径があまり小さくなると超常磁性が発現して保磁力を示さないようになることが知られており、短軸径にも限界がある。また、飽和磁化量も金属磁性粉末粒子の大きさに密接に関係している。現在使用されている金属磁性粉末は、還元したままの金属では大気中で安定に取り扱うことが出来ないため、膜厚20～30Åの酸化皮膜を粒子の表面に形成させており、しかもこの酸化皮膜は、飽和磁化量への寄与は小さいことが知られている。また、この酸化皮膜の厚さは粒子が細かくなっても、大きい粒子と同じ厚さが必要である。従って、粒子を細かくしていくと磁性材に占める酸化皮膜の割合が大きくなり、同じ組成の金属磁性粉末では粒子が細かくなると飽和磁化量が小さくなってしまうこととなる。

【０００３】一方、磁気記録媒体のノイズの改良には、磁性材粒子を微細化することが有効なことが知られている。即ち、磁気記録媒体の単位面積当たりに入る粒子数を多くすることによりノイズの低減が達成出来ることが一般に知られている。粒子の大きさを表す方法の一つとして比表面積があり、磁性材粒子の比表面積とノイズの関係が報告されている（例えば、社団法人　日本磁気メディア工業会発行、磁気メディア技術マニュアルNo.３「磁気記録の原理」中の図４－４，５）。磁性材の比表面積が大きくなると直線的にノイズが低減しているが、比表面積があまり大きくなると分散、パッキング性に問題を生じるので比表面積を大きくすることは好ましくない場合もある。別の見方として、金属磁性粉末の結晶子サイズとノイズの関係も知られており、結晶子サイズが小さいほどノイズが小さくなる。従って、ノイズを小さくする要件は、微細な粒子で、比表面積が分散、パッキング性に悪影響を及ぼさない程度に大きく、結晶子サイズが小さいものである。金属磁性粉末の酸化安定性は、磁気記録媒体の保存安定性と関係するので現在使用されている金属磁性粉末よりも大幅な改良が望まれている。酸化安定性の評価は高温、高湿の雰囲気中に金属磁性粉末を一定期間静置しておき、取り出した後の飽和磁化量を測定し、テスト前の飽和磁化量と比較して行われる。金属磁性粉末粒子が細かくなると、一般的には表面活性が増して酸化されやすくなり安定性は悪くなる傾向がある。また、金属磁性粉末の分散性、パッキング性は、粉

末粒子の形状、粒子表面の化学的、物理的性質により変わると考えられている。形状については、枝別れ、空孔等のない単分散粒子が好ましいとされている。しかしながら、微粒子になるほど分散性、パッキング性は難しくなる。

【０００４】

【発明が解決しようとする課題】近年の磁気記録再生装置の高性能化に伴い、使用する磁気記録媒体の高出力化、低ノイズ化、特に記録波長のより短波長領域での高出力化の要望が強く、使用する金属磁性粉末も、微細で、保磁力が高く、飽和磁化量も大きく、且つ分散性、パッキング性、酸化安定性に優れるものが望まれている。しかしながら、従来の技術では、これらの項目を満足するような金属磁性粉末は得られていない。本発明はこの問題を解決することを目的としたものである。

【０００５】

【課題を解決するための手段】前記従来技術の問題は、以下の本発明によって達成できる。

【０００６】本発明は、第一に、Ni,Co 及び１種以上の稀土類元素並びにSiとAlの化合物を含有する、長軸径が0.05～0.3 μm 、結晶子サイズが130 ～170 Å、比表面積が45～70m²/gの、鉄を主成分とする金属磁性粉であって、保磁力(Hc)が1600～20000e で、且つ温度60℃、相対湿度90％の雰囲気に７日間静置した後の飽和磁化量（σs)が100emu/g以上であることを特徴とする金属磁性粉末である。第二に、比表面積が60～130m²/g であるα-FeOOHにNi,Co,Al,Si 及び１種以上の稀土類元素の化合物を被着させ（但し、既にα-FeOOHに必要量含有されている場合を除く）、非還元性雰囲気下で熱処理し、次いで還元性ガスで還元することを特徴とする上記金属磁性粉末の製造方法である。

【０００７】本発明の金属磁性粉末は、第一鉄塩とアルカリを混合した水懸濁液に、酸化性ガスを吹き込むことによって得られるオキシ水酸化鉄を出発原料とする。このオキシ水酸化鉄の種類としては、α-FeOOHが好ましく、その製法としては、第一鉄塩を水酸化アルカリで中和して$Fe(OH)_2$ の水懸濁液とし、この懸濁液に酸化性ガスを吹き込んで針状のα-FeOOHとする第一の製法がある。一方、第一鉄塩を炭酸アルカリで中和して$FeCO_3$ の水懸濁液とし、この懸濁液に酸化性ガスを吹き込んで紡錘状のα-FeOOHとする第二の製法がある。これらの方法で用いる第一鉄塩としては、塩化第一鉄、硝酸第一鉄、硫酸第一鉄のいずれを使用してもよい。また、第一の製法で用いる水酸化アルカリとしては、水酸化カリウム、水酸化ナトリウム、アンモニア水が使用可能である。また、炭酸アルカリとしては、炭酸ナトリウム、重炭酸ナトリウム、炭酸アンモニウム等が使用可能である。上記第一の製法を用いて、微細で枝分かれがなく、分散性、パッキング性に優れる金属磁性粉末を得るのに適したα-FeOOHを製造する条件は、第一鉄塩の中和に必要なアルカリ量の２～10倍量を使用し、アルカリ濃度の高い条件で$Fe(OH)_2$の酸化反応を進めることである。アルカリ濃度の高い領域での反応は枝分かれのない粒子を得るのに必要な条件である。また、粒子の大きさのコントロールは、一般に知られているように、反応温度、酸化性ガスの吹き込み量のコントロールで可能であるが、その他第一鉄塩中にNi,Co,Al,Si 等の金属塩を存在させておき、これをアルカリで中和してから酸化反応を進めることでも粒子の寸法をコントロールすることが可能である。第二の製法では、生成する紡錘状のα-FeOOHは枝分かれがなく、粒度の揃った微細な粒子が得やすい。また第二の製法における粒子の大きさのコントロールは、水懸濁液中の鉄の濃度、反応温度、酸化性ガスの吹き込み量を変化させることにより可能である。また、第一の製法と同じようにNi,Co 等の添加によっても粒子の形状をコントロールすることが出来る。

【０００８】以下、第一の製法で得た針状のα-FeOOHを原料とする本発明の金属磁性粉末の製造方法を例として説明する。先ず、第一鉄塩を中和に必要な量の２倍量以上の水酸化アルカリで中和して、$Fe(OH)_2$ のアルカリ懸濁液とし、これに酸化性ガスを吹き込んで針状のα-FeOOHを得る。この時、α-FeOOHの針状比及び形状をコントロールするためにNi,Co,Zn,Cr,Mn,Zr,Al,Si,P,Ba,Ca,Mg,Cu,Sr,Ti,Mo,Ag,稀土類元素等の金属をドープしておくことができる。これらの異種金属の添加方法は、第一鉄塩中に均一に混合しておいても良く、また反応の途中で添加しても良い。添加量については所望する形状、大きさにより、経験的に決められる。なお、本方法では、第一鉄塩をアルカリで中和して$Fe(OH)_2$ の懸濁液を生成させ、これを酸化してα-FeOOHを製造するのであるが、この時使用するアルカリ量を中和当量よりも少なくとも２倍以上使用することにより金属磁性粉末とした時に保磁力の高い原料α-FeOOHが得られる。このようなアルカリの過剰量は、多ければ多いほどα-FeOOHの枝分かれは少なくなるが、10倍以上加えてもそれ以上の効果は発現しないため、10倍以上の過剰量を加える反応は効率的でない。また、本発明の金属磁性粉末を得るのに必要なα-FeOOH粒子の大きさは、その比表面積値が60～130m²/gの範囲となる大きさとするのが必要である。この比表面積が60m²/g以下では、粒子が大きすぎて高保磁力は得られないし、単波長領域用の磁性材料として好ましくない。また、比表面積が130m²/g 以上では、粒子が細かくなりすぎ超常磁性が発現するのか、高保磁力は得られないし、また粒子の不揃いによるものか保磁力分布の広いものになってしまう。次に、Ni,Co,Zn,Cr,Mn,Zr,Al,Si,P,Ba,Ca,Mg,Cu,Sr,Ti,Mo,Ag,稀土類元素等がドープされているかまたはドープされていないα-FeOOHに、Ni,Co,Al,Si 及び稀土類元素の１種以上を含有させるのであるが、含有させる方法としては、各種金属塩を酸またはアルカリで中和して、粒子表面上に水酸化物の微小な結晶

の膜として被着する方法が一般的である。Ni,Co,稀土類元素は、α-FeOOHを生成させる反応で必要量ドープされている場合は、新たにα-FeOOH粒子の表面に被着させなくてもよい場合もあるが、これらの元素を多量に含有させる必要がある場合は、ドープされる量に限界があるので、さらに表面にこれらの元素を被着させる。なお、金属磁性粉末中の各金属元素の含有量は以下の範囲が好ましい。以下の数値は鉄を100 としたときの各金属の重量比である。

Ni＝0.3～8.0
Co＝3.0～45.0
Al＝0.5～8.0
Si＝0.5～8.0
稀土類元素＝0.2～10.0
但し、Al＋Si＝2.0～15.0

稀土類元素はLa,Ce,Pr,Nd,Sm,Gd,Dy,Yのうちの少なくとも１種類であり、これらの組合せでも有効である。添加する金属は、塩化物、硫酸塩、硝酸塩等の水溶性塩の使用が便利である。Siの場合はメタ珪酸ソーダ、オルト珪酸ソーダ、水ガラスが使用出来る。被着の順序は、まず合金化して金属磁性粉末の磁気特性をコントロールするNi及びCoを先に被着し、次に熱による粒子の焼結を防止するAlとSiを後半に被着するのが好ましい。稀土類元素の場合はα-FeOOH粒子を生成する時にドープしておいても、あるいはNi及び／又はCoを被着する時に同時に被着しても保磁力を高める効果がある。Al及び／又はSiを被着する時に被着しても効果はあるが、内部に存在させた方が効果は大きい。

【０００９】次に、上記各金属を所定量被着した後、これを十分に水洗して乾燥し、非還元性雰囲気中で、300～800 ℃の温度で熱処理をする。熱処理温度が300 ℃以下では、α-FeOOHが脱水して生じたα-Fe_2O_3粒子中の空孔が多くなり、その結果、還元後の金属磁性粉末の特性が劣ることとなる。また、熱処理温度が800 ℃以上の温度では、α-Fe_2O_3粒子の融解が始まり粒子の形状が変化したり、あるいは焼結が進行し、その結果得られた金属磁性粉末の特性は劣化する。次に、熱処理後の金属磁性粉末を水素ガス気流下で300 ～600 ℃の温度で還元し、公知の方法で粒子の表面に酸化皮膜を形成させて金属磁性粉末を得る。

【００１０】

【作用】本発明の金属磁性粉末は、長軸径0.05～0.3 μm 、結晶子サイズ130 ～170 Å、比表面積45～70m^2/gと微細でありながら、保磁力が1600～2000 Oe と高く、飽和磁化量も高く、且つ温度60℃、相対湿度90％の雰囲気に７日間静置した後の飽和磁化量が100emu/g以上と酸化安定性に優れ、分散性、パッキング性にも優れるものである。従来の技術（例えば、特公昭62－44842 号公報）においてもCo及び稀土類元素とAlまたはSiを含む金属磁性粉末が提案されているが、本発明の金属磁性粉末は、従来の提案の金属磁性粉末に比較して大幅に特性の向上したものである。当該提案との技術的差異は、本発明ではNiの添加及びAlとSiの共存を必須条件としたことにある。Niの添加の効果はAl,Si,稀土類元素等を含むα-FeOOHを加熱処理する時に生成する難還元性の複酸化物中のFe分の還元を促進する働きである。このことにより、還元時の焼結を防止する効果のあるAl,Si,稀土類元素を多量に使用出来るのである。Niの添加量はあまり少ないと還元促進効果は得られず、またあまり多量では保磁力及び飽和磁化量を低下させるので好ましくない。AlとSiの共存は、高保磁力で、分散性、パッキング性に優れ、酸化安定性にも優れる金属磁性粉末を得るのに必須である。即ち、Al単独では焼結防止効果が不十分なためか高保磁力の金属磁性粉末は得られないが、反面分散性、パッキング性に優れ、且つ酸化安定性にも優れるものが得られる。一方、Si単独ではAl単独の場合と逆であり、高保磁力は得やすいが、分散性、パッキング性、酸化安定性はAlの場合と比較してやや劣るものが得られる。従って、AlとSiを共存させることにより、両方の良い点が出現して、高保磁力で分散性、パッキング性、酸化安定性に優れるものが得られるのである。また、さらに稀土類元素は焼結防止効果を有するので、これを含有する場合は、AlとSiだけの場合よりもさらに効果が向上する。このことにより、粒子の形状が原料α-FeOOHの形状を良く保持し、高保磁力、高角型比で結晶子サイズの小さいものが得られる。このような効果を発現する理由は明らかではないが、得られた金属磁性粉末粒子の表面が稀土類元素を添加しなかったものに比較して滑らかであり、空孔も少なくまた折れた粒子も少ないことから、α-FeOOH表面に被着した場合にAlやSiの場合よりも緻密な膜が生成し、熱処理、還元等の体積変化を伴う反応の場合も形骸粒子の体積縮小に追随出来るのではないかと考えられる。以上述べたように、本発明の金属磁性粉末は、高密度磁気記録媒体用の磁性材として好適な、高保磁力、高飽和磁化量、小さい結晶子サイズであり、分散性、パッキング性、酸化安定性に優れるものである。

【００１１】

【実施例】以下に実施例を挙げて本発明を更に詳しく説明するが、本発明はこれらに限定されるものではない。

実施例－１

窒素ガスを流して酸化性ガスを追い出した容器に、2.0mol/lの塩化第一鉄水溶液４リットルと0.23mol の塩化ニッケルを良く混合し、これに2.5mol/lの水酸化ナトリウム水溶液20リットルを攪拌しながら混合して懸濁液を得た。次に、この懸濁液を40℃に保持しつつ10リットル／分の空気を２時間吹き込んで、ニッケルがドープされた針状のα-FeOOHを得た。このものの比表面積を測定したところ75m^2/gであった（表１参照）。次に、α-FeOOHを含有する懸濁液を濾過し、得られた残渣（α-FeOOH）を水洗した後、40リットルの蒸留水中に分散させて懸濁液

とし、この懸濁液を撹拌しながら、塩化コバルト0.76molと塩化ランタン0.065molとを２リットルの蒸留水に溶解した液を徐々に加え、30分間良く撹拌した。撹拌後、この懸濁液中に水酸化ナトリウム水溶液を徐々に添加して懸濁液のpHを８に調整した。次いでこの懸濁液中に、0.5molの塩化アルミニウムを蒸留水２リットルに溶解した水溶液を添加して30分間良く撹拌した。さらにこの懸濁液に、Si分として13.4g を含む水ガラス水溶液を徐々に加えた後、懸濁液のpHを８とするのに必要な量の水酸化ナトリウム水溶液を添加して十分に撹拌した。次に、この懸濁液を濾過し、得られた残渣を水洗して120 ℃の乾燥器中で乾燥し、乾燥ケーキとした。さらにこの乾燥ケーキを窒素雰囲気中で600 ℃で２時間熱処理し、次いで水素ガス流下480 ℃で４時間還元した。還元後、ガスを窒素ガスに変え、温度を室温まで下げた後、窒素ガス流中に空気を徐々に流して常法により安定化処理を行った。以上の操作により得られた金属磁性粉末について、その長軸径および結晶子サイズ、比表面積、磁気特性を測定した結果、長軸径0.24μm 、結晶子サイズ158Å、比表面積55.6m²/g、保磁力Hc 1783 Oe、飽和磁化量σs 128.4emu/gであった。また、この金属磁性粉末の粒子を透過電子顕微鏡で観察したところ、焼結がなく、粒子中にも空孔の少ないものであった。なお、上記各測定値の測定法は以下の通りである。比表面積は、いわゆる窒素測定法を用いて測定し、磁気特性については、詰率0.85g/ccの条件で保磁力Hc、飽和磁化量σs を測定した。また、粉末粒子の長軸径については、透過電子顕微鏡で撮影した金属磁性粉末の写真中から、ランダムに200 個の粒子を抽出し、これら200 個の粒子の平均長軸径で示した。結晶子サイズについては、Ｘ線回折装置で得られた $Fe_{(110)}$ の回折ピークの半価幅、２Θ値から次式により求めた。

$$D_{(110)} = K\lambda / \beta_{cos}\,\Theta$$

Ｋ；シェラー定数(0.9)

λ；照射Ｘ線の波長

β；回折ピークの半価幅（真値に補正して用いる）

Θ；回折角

また、本実施例で得られた金属磁性粉末の酸化安定性を評価するため、この金属磁性粉末を、温度60℃、相対湿度90％の雰囲気中に７日間静置した後、その飽和磁化量を測定した。結果を表２に示す。また、本実施例で得られた金属磁性粉末を高密度磁気記録媒体用磁気テープの磁性材として用いた場合の磁気特性および分散性、パッキング性を評価するため、以下に示す操作に従ってテープ化し、その磁気特性を測定すると共に、このテープ化のBrの数値及び角型比より、分散性、パッキング性を評価した。またテープのS.F.D.値より金属磁性粉末の保磁力分布を評価した。

テープ化の方法

金属磁性粉末100gとポリウレタン樹脂11g 、塩化ビニル－酢酸ビニル共重合体7.75g 、トルエン／メチルエチルケトン／シクロヘキサノン＝１／１／１の混合溶液345gと混合し、サンドグラインダーで５時間分散を行って磁性塗料を作製した。この塗料に架橋剤としてコロネートＬ（日本ポリウレタン（株）製）を2.5g添加した後、ポリエステルフィルムに塗布し、3000ガウスの磁界を印加して、50℃の温度で乾燥した。次いで、乾燥後のフィルムに対し、80℃、線圧200kg/cmでカレンダー処理を行い、60℃で24時間熟成を行った後、テープ状にカットした。次に、得られたテープについて、ＶＳＭ磁力計を用いて最大印加磁場５ｋOeで磁気特性を測定した。結果を表３に示す。

【００１２】実施例－２

窒素ガスを流して酸化性ガスを追い出した容器に、2.0mol/lの塩化第一鉄水溶液４リットルと、0.12mol の塩化ニッケルを良く混合し、これに2.5mol/lの水酸化ナトリウム水溶液20リットルを撹拌しながら混合した。この懸濁液に水ガラスをSi分として0.9gになる量を添加して良く撹拌した後、この懸濁液を40℃に保持しつつ10リットル／分の空気を２時間吹き込み、ニッケルとSiとがドープされた微粒子の針状α-FeOOHを得た。このものの比表面積を測定したところ92m²/gであった（表１参照）。次に、上記α-FeOOHを含有する懸濁液を濾過し、得られた残渣を水洗した後、40リットルの蒸留水に分散させて懸濁液とし、この懸濁液を撹拌しながら、1.5molの塩化コバルトと0.12mol の塩化ニッケル及び0.09mol の塩化ネオジウムとを蒸留水２リットルに溶解した水溶液を加え、30分間良く撹拌し、撹拌後水酸化ナトリウム水溶液を徐々に添加して懸濁液のpHを８に調整した。次いで、上記懸濁液に0.5molの塩化アルミニウムを蒸留水２リットルに溶解した水溶液を添加して30分間良く撹拌し、さらにこの懸濁液に、Si分として5.8gを含む水ガラス水溶液を徐々に加えた。次に、懸濁液のpHを８にするのに必要な量の水酸化ナトリウム水溶液を添加して充分に撹拌した。以下、実施例－１と同様に処理して金属磁性粉末を得た。また、得られた金属磁性粉末について、実施例－１と同様にして、その長軸径および結晶子サイズ、比表面積、磁気特性の測定、並びに酸化安定性の評価を行った。結果を表２に示す。また、本実施例で得られた金属磁性粉末を高密度磁気記録媒体用磁気テープの磁性材として用いた場合の磁気特性等を実施例－１と同様にして評価した。結果を表３に示す。

【００１３】実施例－３～６

α-FeOOHを実施例－２とほぼ同じ条件で４回製造してそれぞれを実施例－３，４，５，６とし、各々について比表面積を測定した。結果を表１に示す。次に、これら実施例－３，４，５，６のα-FeOOHに被着処理を行う際、用いたNi,Co,Al,Si の量を表１に示すように変化させ、また稀土類元素の種類を同表に記載のごとく変化させた以外は、実施例－１と同様に処理して実施例－３～６の

金属磁性粉末を得た。得られた上記実施例－３～６の金属磁性粉末について、実施例－１と同様にして、その長軸径および結晶子サイズ、比表面積、磁気特性の測定、並びに酸化安定性の評価を行った。結果を表２に示す。また、実施例－３～６で得られた金属磁性粉末のうち、実施例５の金属磁性粉末について、高密度磁気記録媒体用磁気テープの磁性材として用いた場合の磁気特性等を実施例－１と同様にして評価した。結果を表３に示す。以下の実施例、比較例においても実施例－１と同様に評価した。

【００１４】実施例－７

α-FeOOHにドープする金属をニッケルとネオジウムにした以外は、実施例－２と同様に処理して金属磁性粉末を得た。

実施例－８

窒素ガスを流して酸化性ガスを追い出した容器に、2.0mol/lの塩化第一鉄水溶液４リットルと、0.5mol/lの炭酸ナトリウム水溶液20リットルを仕込み、よく混合して白色の炭酸鉄を生成させ懸濁液とした。この懸濁液を50℃に保持しつつ撹拌しながら、10リットル／分の空気を２時間吹き込んで紡錘状のα-FeOOHを得た。このものの比表面積値を測定したところ85m²/gであった（表１参照）。上記紡錘状のα-FeOOHを含有する懸濁液を濾過し、得られた残渣を水洗した後、この残渣を40リットルの蒸留水に分散させて懸濁液とし、この懸濁液中に、0.3molの塩化ニッケル、0.76molの塩化コバルト、0.015molの塩化ネオジムを３リットルの蒸留水に溶解した水溶液を、撹拌しながら徐々に添加してさらに30分間よく撹拌した。次いで水酸化ナトリウム水溶液を添加して懸濁液のpHを８に調整した。次いで、この懸濁液に、0.5molの塩化アルミニウムを２リットルの蒸留水に溶解した水溶液を添加して30分間良く撹拌した。さらにこの懸濁液に、Si分として９ｇ の量になるように水ガラス水溶液を徐々に添加した後、水酸化ナトリウム水溶液を添加して懸濁液のpHを８に調整した。次に、この懸濁液を濾過し、得られた残渣を水洗して120 ℃の乾燥器中で乾燥し、乾燥ケーキとした。次にこの乾燥ケーキを窒素雰囲気中550 ℃で２時間熱処理し、次いで水素ガス流下480 ℃で４時間還元した。還元後、ガスを窒素ガスに変え、温度を室温まで下げた後、窒素ガス流中に空気を徐々に流して常法により安定化処理を行った。この得られた金属磁性粉末の粒子を透過電子顕微鏡で観察したところ、長軸径0.12μm の紡錘状を呈した粒子であった。

実施例－９

窒素ガスを流して酸化性ガスを追い出した撹拌機付容器に、2.0mol/lの硫酸第一鉄水溶液４リットルと、1.5molの塩化コバルトをよく混合し、これに０．７ｍｏｌ／ｌの炭酸ナトリウム水溶液２０リットルと２mol/lの水酸化ナトリウム水溶液１リットルを添加して30分間良く撹拌し懸濁液とした。次に、この懸濁液を40℃に保持しつつ10リットル／分の空気を２時間吹き込み、コバルトがドープされた紡錘状のα-FeOOHを得た。上記紡錘状のα-FeOOHを含有する懸濁液を濾過し、得られた残渣を水洗し、乾燥した後、実施例－８と同様の処理を行って紡錘状の金属磁性粉末を得た。

実施例－10

窒素ガスを流して酸化性ガスを追い出した撹拌機付容器に、2.0mol/lの硫酸第一鉄水溶液４リットルと、0.75mol の塩化コバルトを良く混合し、これに0.7mol/lの炭酸アンモニウム水溶液20リットルを添加して30分間良く撹拌した。この懸濁液を45℃に保持しつつ、15リットル／分の空気を２時間吹き込み、コバルトがドープされた紡錘状のα-FeOOHを得た。上記紡錘状のα-FeOOHを含有する懸濁液を濾過し、得られた残渣を水洗し、乾燥した後、実施例－８と同様に処理して紡錘状の金属磁性粉末を得た。

実施例－11

実施例－８と添加金属量が異なるだけで、他は同じ条件で処理して紡錘状の金属磁性粉末を得た。

【００１５】比較例－１

稀土類元素のランタンを添加しなかった以外は、全て実施例－１と同様に処理して針状の金属磁性粉末を得た。なお、表２および表３からも明らかなように、比較例－１の金属磁性粉末は、実施例－１で得られた金属磁性粉末に比較して保磁力が低く、結晶子サイズも大きいものであった。

比較例－２

コバルトを添加しなかった以外は全て実施例－２と同様に処理して針状の金属磁性粉末を得た。なお、表２および表３からも明らかなように、比較例－２の金属磁性粉末は、実施例－２に比較して保磁力が低くなり、金属磁性粉末の温度60℃、相対湿度９０％中に７日間静置した後の飽和磁化量が低くなり、酸化安定性が不充分なものであった。

比較例－３

ニッケルを添加しなかった以外は実施例－８と同様に処理して紡錘状の金属磁性粉末を得た。なお、表２からも明らかなように、比較例－３の金属磁性粉末は、実施例－８に比較して保磁力、飽和磁化量、角型比共に低いものとなった。

比較例－４

稀土類元素を添加しなかった以外は全て実施例－９と同様に処理して紡錘状の金属磁性粉末を得た。なお、表２および表３からも明らかなように、比較例－４の金属磁性粉末は、実施例－９に比較して保磁力、角型比が低下し、結晶子サイズが著しく大きいものであった。

比較例－５

実施例－８と同じ方法で原料の紡錘状α－ＦｅＯＯＨを製造し、Siを添加せずに処理して紡錘状の金属磁性粉末としたものである。なお、表２からも明らかなように、

比較例－５の金属磁性粉末は、Siを添加した物に比較して保磁力が著しく低いものであった。

【００１６】比較例－６

実施例－８と同じ方法で原料の紡錘状α-FeOOHを製造し、Alを添加せずに処理して紡錘状の金属磁性粉末としたものである。なお、表２および表３からも明らかなように、Alを添加したものに比較して保磁力は高いものになるが、粉末の酸化安定性とテープにしたときの分散性が劣るものであった。

【００１７】

【表１】

添加金属の種類と添加量

No.	ドープ金属種類と添加量	添加金属量（Feを100 とした時の重量比） Ni	Co	Al	Si	稀土類	α-FeOOHの比表面積(m^2/g)
実施例－１	Ni=3.0	3.0	5.0	3.0	2.0	La=2.0	75.0
実施例－２	Ni=1.5 Si=0.2	3.0	20.0	3.0	1.5	Nd=3.0	92.0
実施例－３	Ni=1.5 Si=0.2	1.5	10.0	4.0	1.0	Dy=1.0	84.0
実施例－４	Ni=1.5 Si=0.2	5.0	10.0	2.5	2.5	Pr=1.0 Nd=1.0	90.0
実施例－５	Ni=1.5 Si=0.2	3.0	15.0	0.7	3.5	Nd=3.5	98.0
実施例－６	Ni=1.5 Si=0.2	3.0	15.0	4.5	0.7	Y =3.0	95.0
実施例－７	Ni=1.5 Nd=0.5	2.0	10.0	3.0	1.5	Nd=2.0	115.0
実施例－８		4.0	10.0	3.0	2.0	Nd=0.5	85.0
実施例－９	Co=20.0	5.0	40.0	2.0	1.5	Dy=3.5	90.0
実施例－10	Co=10.0	3.0	20.0	2.5	1.0	Nd=5.0	105.0
実施例－11		3.0	5.0	4.0	2.0	Nd=2.0	80.0
比較例－１	Ni=3.0	3.0	5.0	3.0	2.0	無添加	77.0
比較例－２	Ni=1.5 Si=0.2	3.0	無添加	3.0	1.5	Nd=3.0	92.0
比較例－３		無添加	10.0	3.0	2.0	Nd=0.5	86.0
比較例－４	Co=20.0	5.0	40.0	2.0	1.5	無添加	90.0
比較例－５		4.0	10.0	4.5	無添加	Nd=2.0	86.0
比較例－６		4.0	10.0	無添加	4.5	Nd=2.0	83.0

【００１８】

【表２】

金属磁性粉末の磁気特性とその他特性

No.	磁気特性（測定磁場=10KOe）			平均長軸径（μm）	結晶子サイズ（Å）	比表面積（m^2/g）	高温高湿7日後のσs
	Hc(Oe)	σs(emu/g)	σr/σs				
実施例－1	1,783	128.4	0.520	0.24	158	55.6	102.8
実施例－2	1,926	145.7	0.535	0.17	163	57.4	115.0
実施例－3	1,628	136.5	0.515	0.16	157	58.5	110.5
実施例－4	1,820	135.0	0.523	0.14	161	60.4	107.1
実施例－5	1,964	139.2	0.540	0.18	167	54.5	106.4
実施例－6	1,651	137.4	0.516	0.15	155	62.3	111.5
実施例－7	1,718	132.8	0.518	0.11	158	58.0	111.4
実施例－8	1,720	134.0	0.498	0.12	147	59.5	108.6
実施例－9	1,825	149.3	0.502	0.08	165	48.7	121.8
実施例－10	1,684	142.3	0.500	0.10	156	58.5	111.0
実施例－11	1,625	126.8	0.496	0.14	143	60.3	104.3
比較例－1	1,520	133.5	0.492	0.21	172	52.5	98.5
比較例－2	1,708	122.5	0.502	0.18	158	58.8	84.7
比較例－3	1,587	124.7	0.477	0.13	155	62.4	92.4
比較例－4	1,494	158.7	0.469	0.07	196	43.5	120.7
比較例－5	1,460	135.1	0.472	0.11	140	52.0	108.8
比較例－6	1,890	131.4	0.505	0.14	145	54.3	88.5

【００１９】
【表３】

テ　ー　プ　特　性

No.	テープの特性（測定磁場＝5kOe）			
	Hc(Oe)	Br(G)	角型比	S.F.D.
実施例－1	1,802	3,052	0.92	0.41
実施例－2	1,946	3,585	0.90	0.43
実施例－5	2,094	3,317	0.91	0.42
実施例－7	1,763	3,162	0.87	0.45
実施例－8	1,785	3,426	0.86	0.46
実施例－9	1,923	3,720	0.87	0.48
実施例－10	1,727	3,612	0.86	0.45
比較例－1	1,544	2,680	0.81	0.56
比較例－2	1,753	2,680	0.84	0.49
比較例－4	1,514	3,418	0.73	0.66
比較例－6	1,905	2,475	0.74	0.53

【００２０】

【発明の効果】以上述べた実施例と比較例との比較において明らかなように、本発明の金属磁性粉末は、微粒子でありながら、高密度磁気記録媒体用の磁性粉に要求される高保磁力、高飽和磁化量を有し、保磁力分布(S.F.D.)、酸化安定性に優れ、さらに分散性にも優れるものである。

フロントページの続き

(72)発明者　守谷　好美
群馬県渋川市金井425番地　関東電化工業株式会社研究開発センター内

(72)発明者　遠藤　盈
群馬県渋川市金井425番地　関東電化工業株式会社研究開発センター内